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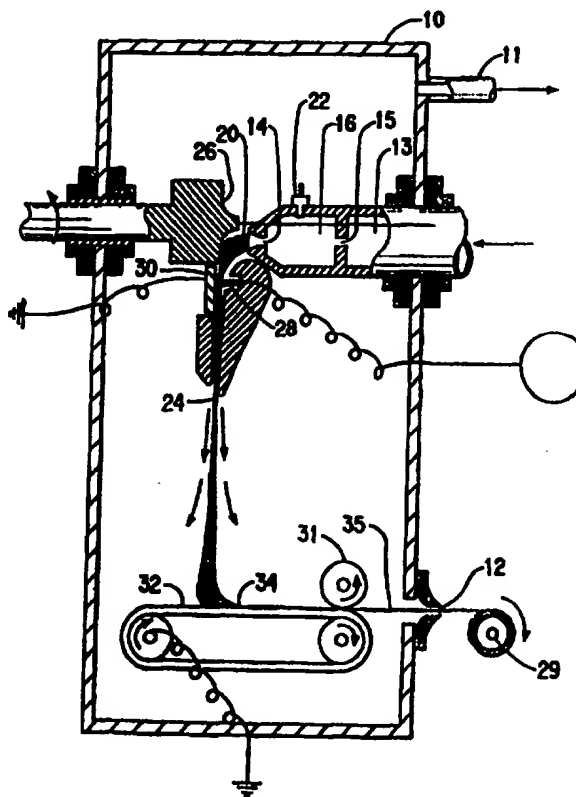
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(54) Title: FLASH-SPUN SHEET MATERIAL

(57) Abstract

This invention relates to improved synthetic sheet material useful in protective apparel, which material has a hydrostatic head pressure of at least about 75 cm of water, a Gurley Hill Porosity of less than about 15 seconds, and a Handle-o-meter stiffness of less than 28 mN/g/m². The sheet material also demonstrates excellent bacteria and particulate barrier properties. The improved sheet material properties are obtained by reducing the ratio of polymer to the spin agent during spinning and by increasing the temperature of the spin solution such that smaller and less cohesive fibers are spun and laid down to form the fabric.



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FLASH-SPUN SHEET MATERIAL

Field of the Invention

This invention relates to sheets or fabrics suited for protective apparel as well as to other end use applications in which a sheet or fabric
5 must demonstrate good liquid and particulate barrier properties as well as a high degree of breathability.

Background of the Invention

Protective apparel includes coveralls, gowns, smocks and other garments whose purpose is either to protect a wearer against exposure to
10 something in the wearer's surroundings, or to protect the wearer's surroundings against being contaminated by the wearer. Examples of protective apparel include suits worn in microelectronics manufacturing cleanrooms, medical suits and gowns, dirty job coveralls, and suits worn for protection against liquids or particulates. The particular applications for
15 which a protective garment is suitable depends upon the composition of the fabric or sheet material used to make the garment and the way that the pieces of fabric or sheet material are held together in the garment. For example, one type of fabric or sheet material may be excellent for use in hazardous chemical protection garments, while being too expensive or uncomfortable
20 for use in medical garments. Another material may be lightweight and breathable enough for use in clean room suits, but not be durable enough for dirty job applications.

The physical properties of a fabric or sheet material determine the protective apparel applications for which the material is suited. It has been
25 found desirable for a wide variety of protective garment applications that the material used in making the protective garment provide good barrier protection against liquids such as body fluids, paints or sprays. It is also desirable that the material used in making protective apparel block the passage of fine dirt, dust and fiber particles. Another group of desirable
30 properties for fabrics or sheet materials used in protective apparel is that the material have enough strength and tear resistance that apparel made using the sheet material not lose its integrity under anticipated working conditions.

It is also important that fabrics and sheet materials used in protective garments transmit and dissipate both moisture and heat so as to permit a wearer to perform physical work while dressed in the garment without becoming excessively hot and sweaty. Finally, most protective garment materials must have a manufacturing cost that is low enough to make the use of the material practical in low cost protective garments.

A number of standardized tests have been devised to characterize materials used in protective garments so as to allow others to compare properties and make decisions as to which materials are best suited to meet the various anticipated conditions or circumstances under which a garment will be required to serve. The strength and durability of sheet materials for apparel have been quantified in terms of tensile strength, tear strength and elongation. The primary test used for characterizing liquid barrier properties is a test of resistance to passage of water at various pressures known as the hydrostatic head resistance test. Particulate barrier properties are measured by bacterial barrier tests and particle penetration tests.

Thermal comfort of fabrics and sheet materials has traditionally been presumed to correspond to the tested moisture vapor transmission rate (MVTR) of the material. However, MVTR is determined under static laboratory conditions, which measure vapor transported by molecular diffusion only. MVTR test results have not proved to be an entirely reliable means of predicting an apparel sheet material's comfort under actual dynamic workplace conditions.

Another test method that has attempted to characterize the thermal comfort of apparel materials is the sweating hot plate test which measures a material's wet and dry heat transfer properties under conditions that simulate a perspiring human in a warm working environment. According to the hot plate test method, a fabric sample in a controlled humidity environment is placed on a hot plate. Water is injected onto the plate to simulate sweating and a controlled air flow is blown over the exposed fabric surface. The heat flow through the sheet material is measured with and without water injection, and thermal property measurements of dry and wet heat transfer are obtained. The sweating hot

plate test is more fully described in Kawabata et al., "Application of the New Thermal Tester THERMOLABO to the Evaluation of Clothing Comfort," Objective Measurement: Application to Product Design and Process Control, The Machinery Society of Japan, 1985, which is hereby incorporated by
5 reference.

The dry heat transfer measured from the sweating hot plate test can be converted to the more conventional "clo" units of clothing insulation. A greater the "clo" value indicates a greater the resistance to dry heat transport. Accordingly, a fabric with a higher "clo" value will be perceived
10 as less comfortable than a fabric with a lower "clo" value.

Data from the sweating hot plate test can also be used to calculate a moisture permeability index "im", which compare the actual ratio of evaporative to dry heat transfer to the theoretical limit. A higher "im" value means a greater ability to transport moisture vapor through the fabric, which
15 would be expected to make the fabric more comfortable. According to the literature, humans can detect differences of 0.01 "im" units while differences of 0.02 units are manifested in changes in heart rate, skin and body temperature.

The "clo" and "im" values calculated from the sweating hot plate
20 test data for a garment material can be used to calculate the theoretical metabolic activity level that a wearer of a garment made of the material could sustain without overheating. An equation developed by A.H. Woodcock, that is based on a heat balance model and is well known in the art, is used to make the calculation. Woodcock's equation is more fully
25 described in: Woodcock, A.H., "Moisture in Textile Systems, Parts I and II," Textile Research Journal, 32, 1962, pp. 626, 719, which is hereby incorporated by reference. A "Comfort Limit" activity level can be predicted by using Woodcock's heat balance equation and incorporating a factor to allow for a 20% sweat wetted area of the human body (having more
30 than 20% sweat coverage for the human body is considered "uncomfortable"). The "max" limit can be similarly calculated that allows for total 100% sweat coverage of the body (maximum evaporation possible). These values simply provide another way of comparing performance of the

fabrics according to which a higher comfort limit or max limit relates to greater productivity.

While the sweating hot plate test method more closely simulates conditions under which protective apparel is used than does MVTR testing, the hot plate test method has still not proved to be the most reliable predictor of the relative comfort of various sheet materials when used in apparel products. In a study of various apparel sheet materials conducted for DuPont by an independent testing laboratory, it was learned that a material's air permeability was the most reliable predictor of the relative comfort afforded by various fabric and sheet materials worn in protective garments. In this study, identical suits made of five different materials were worn by test subjects under conditions designed to simulate realistic, but stressful, work conditions that might be experienced on a summer day in the midwestern United States (90°F/32°C with 60% relative humidity). The subjects walked on an incline treadmill with a 2.5% grade at a moderate walking speed, which speed was increased 0.1 mph (2.68 m/min) every five minutes. The subject's metabolic rate, heart rate and skin and core temperatures were constantly monitored. When the excess heat generated by the increased walking speed exceeded the cooling capability of the test subject inside the garment, the test subject's core body temperature began to rise, indicating a loss of thermal equilibrium and the beginnings of heat stress.

In this study, it was found that the maximum treadmill speed that could be attained by the test subjects in the various garments before the onset of heat stress did not directly correlate to the MVTR values or the heat transfer values determined by the sweating hot plate test. However, a direct correlation was found between the air flow permeability of the various garment sheet materials and the maximum treadmill speed attained before the loss to thermal equilibrium. The significant contribution that air permeability makes to the thermal comfort of a garment appears to be due to motion induced pumping of air and moisture through the fabric or material. Because molecular diffusion of water vapor (measured by MVTR and hot plate tests) is a relatively slow process, it appears that even small flows of

moisture-laden air through a fabric or sheet material can have significantly more impact on moisture vapor transport through a material. Accordingly, it is important that sheet materials used in protective apparel have a high degree of air permeability without unduly sacrificing other important properties such as strength or barrier.

Tyvek® spunbonded olefin has been in use for a number of years as a material for protective apparel. E. I. du Pont de Nemours and Company (DuPont) makes and sells Tyvek® spunbonded olefin nonwoven fabric. Tyvek® is a trademark owned by DuPont. Tyvek® nonwoven fabric has been a good choice for protective apparel because of its excellent strength properties, its good barrier properties, its light weight, its reasonable level of thermal comfort, and its single layer structure that gives rise to a low manufacturing cost relative to most competitive materials. DuPont has worked to further improve the comfort of Tyvek® fabrics for garments. For example, DuPont markets a Tyvek® Type 16 fabric style that includes apertures to improve breathability. DuPont has also produced water jet softened Tyvek® fabric (e.g., U.S. Patent No. 5,023,130 to Simpson) that is softer and more opened up to enhance comfort and breathability. While both of these materials are indeed more comfortable, the barrier properties of these materials are significantly reduced as a consequence of their increased breathability.

Thus, there is a need for a sheet material suitable for use in protective apparel that has strength, weight and barrier properties at least equivalent to that of the Tyvek® spunbonded olefin nonwoven fabric currently used for protective garments, but that also has significantly improved breathability to enhance the thermal comfort of protective apparel made of the material.

Summary of the Invention

The above and other properties of the present invention are achieved by a synthetic sheet material useful in protective apparel, which material has a hydrostatic head pressure of at least about 75 cm of water, a Gurley Hill Porosity of less than about 15 seconds, and a Handle-o-meter stiffness of less than 28 mN/g/m². Preferably the sheet material has a

hydrostatic head pressure of at least about 90 cm of water and a Gurley Hill Porosity of less than about 12 seconds, and more preferably the Gurley Hill Porosity is less than about 10 seconds.

It is further preferred that the sheet material of the invention have
5 a bacteria spore penetration, measured according to ASTM F1608-95, of less than 5% and a particulate filtration efficiency, measured according to IES standard IES-RP-CC003.2, Section 7.3.1, of at least 95%. The most preferred sheet material has an MVTR-LYSSY, measured according to
ASTM E398-83, of at least 1300 g/m²/day and a basis weight of at least
10 30 g/m².

The sheet material of the preferred embodiment of the invention has a tensile strength in both the machine and cross directions of at least 1250 N/m and a tongue tear in both the machine and cross directions of 250 N/m. The sheet material of the preferred embodiment of the invention
15 is a synthetic material comprised primarily of nonwoven fibers, said sheet material having a hydrostatic head pressure of at least about 75 cm of water, a Gurley Hill Porosity of less than about 15 seconds, and a Handle-o-meter stiffness of less than 28 mN/g/m². Preferably the sheet material is substantially exclusively a unitary sheet of nonwoven fibers.

20 The present invention is further directed to a protective garment comprising a plurality of interconnected sheet material pieces, each of said sheet material pieces comprising a synthetic sheet material having a hydrostatic head pressure of at least about 75 cm of water, a Gurley Hill Porosity of less than about 15 seconds, and a Handle-o-meter stiffness of
25 less than 28 mN/g/m².

The present invention is also directed to a process for flash spinning polymer and forming sheet material therefrom, the improvement comprising mixing the polymer in a pentane spin agent at a ratio of less than about 16% polymer, and emitting the polymer solution through a spin orifice
30 at a temperature of at least about 180°C. An alternative embodiment of the invention is directed to a process for flash spinning polymer and forming sheet material therefrom, the improvement comprising mixing the polymer in a trichlorofluorocarbon spin agent at a ratio of less than about 11%

polymer, and emitting the polymer solution through a spin orifice at a temperature of at least about 185°C.

Brief Description of the Drawings

The invention will be more easily understood by a detailed
5 explanation of the invention including drawings. Accordingly, drawings which are particularly suited for explaining the invention are attached herewith; however, it should be understood that such drawings are for explanation only and are not necessarily drawn to scale.

Figure 1 a schematic cross sectional view of a spin cell
10 illustrating the basic process for making flash-spun nonwoven products; and
Figure 2 is an enlarged cross sectional view of the spinning equipment for flash spinning fiber.

Detailed Description of the Preferred Embodiment

The process for making flash-spun nonwoven products, and
15 specifically Tyvek® spunbonded olefin, was first developed more than twenty-five years ago and put into commercial use by DuPont. U.S. Pat. No. 3,081,519 to Blades et al. (assigned to DuPont), describes a process wherein a solution of fiber-forming polymer in a liquid spin agent that is not a solvent for the polymer below the liquid's normal boiling point, at a
20 temperature above the normal boiling point of the liquid, and at autogenous pressure or greater, is spun into a zone of lower temperature and substantially lower pressure to generate plexifilamentary film-fibril strands. As disclosed in U.S. Pat. No. 3,227,794 to Anderson et al. (assigned to DuPont), plexifilamentary film-fibril strands are best obtained using the
25 process disclosed in Blades et al. when the pressure of the polymer and spin agent solution is reduced slightly in a letdown chamber just prior to flash-spinning.

The term "plexifilamentary" as used herein, means a three-dimensional integral network of a multitude of thin, ribbon-like, film-fibril
30 elements of random length and with a mean film thickness of less than about 4 microns and a median fibril width of less than about 25 microns. In plexifilamentary structures, the film-fibril elements are generally

coextensively aligned with the longitudinal axis of the structure and they intermittently unite and separate at irregular intervals in various places throughout the length, width and thickness of the structure to form a continuous three-dimensional network.

5 Flash spinning of polymers using the process of Blades et al. and Anderson et al. requires a spin agent that: (1) is a non-solvent to the polymer below the spin agent's normal boiling point; (2) forms a solution with the polymer at high pressure; (3) forms a desired two-phase dispersion with the polymer when the solution pressure is reduced slightly in a letdown
10 chamber; and (4) flash vaporizes when released from the letdown chamber into a zone of substantially lower pressure. Depending on the particular polymer employed, the following compounds have been found to be useful as spin agents in the flash-spinning process: aromatic hydrocarbons such as benzene and toluene; aliphatic hydrocarbons such as butane, pentane,
15 hexane, heptane, octane, and their isomers and homologs; alicyclic hydrocarbons such as cyclohexane; unsaturated hydrocarbons; halogenated hydrocarbons such as trichlorofluoromethane, methylene chloride, carbon tetrachloride, dichloroethylene, chloroform, ethyl chloride, methyl chloride; alcohols; esters; ethers; ketones; nitriles; amides; fluorocarbons; sulfur
20 dioxide; carbon dioxide; carbon disulfide; nitromethane; water; and mixtures of the above liquids. Various solvent mixtures useful in flash-spinning are disclosed in U.S. Patent 5,032,326 to Shin; U.S. Patent 5,147,586 to Shin et al.; and U.S. Patent 5,250,237 to Shin (all assigned to DuPont).

 The process for flash-spinning sheets comprised of
25 plexifilamentary film-fibril strands is illustrated in Figure 1, and is similar to that disclosed in U.S. Patent 3,860,369 to Brethauer et al., which is hereby incorporated by reference. The flash-spinning process is normally conducted in a chamber 10, sometimes referred to as a spin cell, which has an exhaust port 11 for exhausting the spin cell atmosphere to a spin agent
30 recovery system and an opening 12 through which non-woven sheet material produced in the process is removed.

 A solution of polymer and spin agent is provided through a pressurized supply conduit 13 to a letdown orifice 15 and into a letdown

chamber 16. The pressure reduction in the letdown chamber 16 precipitates the nucleation of polymer from a polymer solution, as is disclosed in U.S. Patent 3,227,794 to Anderson et al. One option for the process is to include an inline static mixer 36 (see Figure 2) in the letdown chamber 16. A
5 suitable mixer is available from Koch Engineering Company of Wichita Kansas as Model SMX. A pressure sensor 22 may be provided for monitoring the pressure in the chamber 16. The polymer mixture in chamber 16 next passes through spin orifice 14. It is believed that passage
10 of the pressurized polymer and spin agent from the letdown chamber 16 into the spin orifice 14 generates an extensional flow near the approach of the orifice that helps to orient the polymer into elongated polymer molecules. As the polymer passes through the spin orifice, the polymer molecules are further stretched and aligned. When polymer and spin agent discharge from
15 the spin orifice 14, the spin agent rapidly expands as a gas and leaves behind fibrillated plexifilamentary film-fibrils. The spin agent's expansion during flashing accelerates the polymer so as to further stretch the polymer molecules just as the film-fibrils are being formed and the polymer is being cooled by the adiabatic expansion. The quenching of the polymer freezes the linear orientation of the polymer molecule chains in place, which
20 contributes to the strength of the resulting flash-spun plexifilamentary polymer structure.

The gas exits the chamber 10 through the exhaust port 11. The polymer strand 20 discharged from the spin orifice 14 is conventionally directed against a rotating lobed deflector baffle 26. The rotating baffle 26
25 spreads the strand 20 into a more planar web structure 24 that the baffle alternately directs to the left and right. As the spread web descends from the baffle, the web is passed through an electric corona generated between an ion gun 28 and a target plate 30. The corona charges the web so as to hold it in a spread open configuration as the web 24 descends to a moving belt 32
30 where the web forms a batt 34. The belt is grounded to help insure proper pinning of the charged web 24 on the belt. The fibrous batt 34 is passed under a consolidation roll 31 that compresses the batt into a sheet 35 formed with plexifilamentary film-fibril networks oriented in an overlapping multi-

directional configuration. The sheet 35 exits the spin chamber 10 through the outlet 12 before being collected on a sheet collection roll 29.

The sheet 35 is subsequently run through a finishing line which treats and bonds the material in a manner appropriate for its end use. For example, the sheet product may be bonded on a smooth heated roll as disclosed in U.S. Patent 3,532,589 to David (assigned to DuPont) in order to produce a hard sheet product. According to this bonding process, both sides of the sheet are subjected to generally uniform, full surface contact thermal bonding. The "hard structure" product has the feel of slick paper and is used commonly in overnight mailing envelopes, for construction membrane materials such as Tyvek® Homewrap™, in sterile packaging, and in filters. Full surface bonded "hard structure" material is unlikely to be used in apparel applications due to its paper-like feel and drape.

For apparel applications, the sheet 35 is typically point bonded and softened as disclosed in U.S. Patents 3,427,376 and 3,478,141 (both assigned to DuPont) to produce a "soft structure" product with a more fabric like feel. The intent with point bonding is to provide closely spaced bonding points with unbonded fiber therebetween in an aesthetically pleasing pattern. DuPont prefers a point bonding pattern according to which the sheet is contacted by thermal bonding rolls with undulated surfaces that give rise to portions of the fabric having very slight thermal bonding while other portions are more clearly subjected to bonding. After the fabric sheet is bonded, it is subjected to mechanical softening to remove hardness that may have been introduced during bonding. This improves the feel and tactile qualities of the fabric.

It is thought that the full surface bonding of a "hard structure" flash-spun sheet product causes the high surface area plexifilamentary fibers of the sheet to shrink, which in turn causes the pores between the fibers to open up. Accordingly, "hard structure" sheet products generally have higher MVTR's and higher hydrostatic head numbers as compared to "soft structure" sheet products. Thus, when describing physical properties of flash-spun sheet products, it is important to differentiate between hard and soft structure products. Handle-o-meter stiffness measurements can be used

to differentiate hard and soft structure products. For purposes of comparison, such stiffness values are normalized to the basis weight (divided by basis weight).

5 Tyvek® Style 1042B, a hard structure material having a low basis weight of 1.25 oz/yd², has a handle-o-meter stiffness of 1290 mN which can be normalized to 30.4 mN/g/m². Heavier basis weight "hard structure" sheets are expected to be at least as stiff even when normalized as the Style 1042B. The point bonded "soft structure" product Tyvek® Style 1422A, which has a basis weight of 1.2 oz/yd², has a Handle-o-meter stiffness of 10 430 mN. This is a normalized stiffness of 10.6 mN/g/m². The heavier weight "soft structure" Tyvek® Style 1673, with a basis weight of 2.10 oz/yd² and a Handle-o-meter of 1640 mN, has a normalized stiffness of 23.1 mN/g/m². A normalized stiffness of less than about 28 mN/g/m² in a flash-spun sheet is indicative of a "soft structure" product, and a normalized 15 stiffness of less than 25 mN/g/m² will very clearly be a "soft structure" sheet product.

It should be recognized that permeability, MVTR and hydrostatic head properties of a flash-spun sheet or fabric material may each be modified by post spinning treatment such as bonding. However, while 20 excessive bonding can be used to increase the MVTR and hydrostatic head of a flash-spun sheet to a point, such bonding will generally cause other important properties to fall below that which are acceptable. For example, excessive bonding of a flash-spun polyolefin sheet material normally causes the material's opacity to drop below the 85% level that is deemed minimally 25 acceptable for apparel end uses. High bonding levels can also adversely impact a flash-spun sheet material's softness, durability and barrier properties.

Historically, the preferred spin agent used in making Tyvek® flash-spun polyethylene has been the chlorofluorocarbon (CFC) spin agent, 30 trichlorofluoromethane (FREON®-11). FREON® is a registered trademark of DuPont. When FREON®-11 is used as the spin agent, the spin solution has been comprised of about 12% by weight of polymer with the remainder

being spin agent. The temperature of the spin solution just before flashing has historically been maintained at about 180°C.

It has now been found that it is possible to flash-spin finer plexifilamentary fibers that, when laid down and bonded, make a Tyvek® fabric or sheet that is significantly more permeable than the Tyvek® fabric
5 or sheet material produced from a 12% polyethylene/88% FREON®-11 solution at a spin temperature of about 180° C, and with at least equivalent strength and barrier properties. This more permeable material has been found to have great utility in protective garments where increased air
10 permeability improves the comfort of garments made using the material.

Applicants have found that improved fabric sheet permeability can be attained, when flash-spun polyethylene fabric or sheet material is manufactured using a FREON®-11 based spin solution, by reducing the concentration of the polymer in the spinning solution and by raising the
15 temperature at which the spinning solution is maintained prior to flashing. As disclosed in the examples below, reducing the concentration of polyethylene in the FREON®-11 based spin solution to between 9% and 11% of the spin solution and increasing the spinning temperature to between 185° to 195°C has been found to significantly improve the permeability of
20 the bonded fabric material produced while maintaining at least equivalent strength and barrier properties.

Without wishing to be bound by theory, it is presently believed that as the polymer concentration is reduced the average fiber size becomes smaller, and as the solution spin temperature is increased the fibers become
25 less cohesive. The smaller fibers are believed to result in sheet layers with fewer thicker portions therein and with a larger number of smaller pores. However, the sheet appears to have an overall structure that is less cohesive with larger void spaces between the layers in the plane of the sheet. The end result seems to be a sheet that allows more gas and vapor to pass making the
30 material much more permeable without a reduction in barrier properties. The data in Examples 24 and 25 below show that the mean fiber size of the fibers before bonding is smaller for the higher permeability sample spun at a lower polymer concentration and an increased solution temperature (Ex. 25).

Applicants have also found that it is possible to flash-spin a polyethylene fabric or sheet material with improved permeability and with barrier strength properties equivalent to conventional Tyvek® flash-spun polyethylene sheets by flash-spinning the sheet from a hydrocarbon-based spin solution comprised of between 12% and 16% by weight polyethylene and maintained at a temperature of between 185° to 195°C prior to flashing. Such materials are more fully disclosed in the examples below.

Importantly, the more permeable fabric or sheet material of the present invention maintains the strength and durability of conventional Tyvek® flash-spun polyethylene sheets because of the molecular orientation of the polymer in the fibers and because the sheet can be made in a single laydown process with a single polymer. In addition, recyclability and lower cost are built into the uniform flash-spun fabrics or sheet materials of the present invention as compared to the laminated products with which the material of the invention must compete in the marketplace. As used herein, the term "unitary sheet" is used to designate a nonwoven sheet made exclusively of similar fibers of a single polymer, and that is free of laminations or other support structures. Finally, the flash-spun fabric material of the present invention has barrier and strength properties suitable for protective garments at a commercial basis weight of 40.5 g/m² (1.2 oz/yd²) which compares quite favorably to the heavier competitive laminated products which are commercially available at basis weights of 64.5 g/m² (1.9 oz/yd²) and greater.

This invention will now be illustrated by the following non-limiting examples which are intended to illustrate the invention and not to limit the invention in any manner.

EXAMPLES

In the description above and in the non-limiting examples that follow, the following test methods were employed to determine various reported characteristics and properties. ASTM refers to the American Society for Testing and Materials, AATCC refers to the American

Association of Textile Chemists and Colorists, and IES refers to the Institute of Environmental Sciences.

Basis Weight was determined by ASTM D-3776, which is hereby incorporated by reference, and is reported in g/m^2 . The basis weights reported for the examples below are each based on an average of at least twelve measurements made on the sample.

Tensile Strength and Work to Break were determined by ASTM D-1682, Section 19, which is hereby incorporated by reference, with the following modifications. In the test, a 2.54 cm by 20.32 cm (1 inch by 8 inch) sample was clamped at its opposite ends. The clamps were attached 12.7 cm (5 in) from each other on the sample. The sample was pulled steadily at a speed of 5.08 cm/min (2 in/min) until the sample broke. The force at break was recorded Newtons/cm as the breaking tensile strength. The area under the stress-strain curve was the work to break.

Grab Tensile Strength was determined by ASTM D 1682, Section 16, which is hereby incorporated by reference, and is reported in Newtons.

Hydrostatic Head is a measure of the resistance of the sheet to penetration by liquid water under a static load. A 7x7 in (17.78x17.78 cm) sample is mounted in a SDL 18 Shirley Hydrostatic Head Tester (manufactured by Shirley Developments Limited, Stockport, England). Water is pumped against one side of a 102.6 cm^2 section of the sample at a rate of 60 +/- 3 cm/min until three areas of the sample are penetrated by the water. The measured hydrostatic pressure is measured in inches, converted to SI units and given in centimeters of water. The test generally follows ASTM D 583 (withdrawn from publication November, 1976).

Moisture Vapor Transmission Rate (MVTR) is determined by two methods: ASTM E96, Method B, and ASTM E398-83 (which has since been withdrawn), which are hereby incorporated by reference. MVTR is reported in $\text{g/m}^2/24 \text{ hr}$. MVTR data aquired using ASTM E96, Method B is labeled herein simply as "MVTR" data. MVTR data acquired by ASTM E398-83 was collected using a Lyssy MVTR tester model L80-4000J and is identified herein as "MVTR-LYSSY" data. Lyssy is based in Zurich, Switzerland. MVTR test results are highly dependent on the test method used and material type. Important variables between test methods include

pressure gradient, volume of air space between liquid and sheet sample, temperature, air flow speed over the sample and test procedure.

ASTM E96, Method B is a gravimetric method that uses a pressure gradient of 100% relative humidity (wet cup) vs. 55% relative humidity (ambient). ASTM E96, Method B is based on a real time measurement of 24 hours during which time the humidity delta changes and the air space between the water in the cup and the sample changes as the water evaporates.

ASTM E398-83 (the "LYSSY" method) is based on a pressure gradient of 85% relative humidity ("wet space") vs. 15% relative humidity ("dry space"). The LYSSY method measures the moisture diffusion rate for just a few minutes and under a constant humidity delta, which measured value is then extrapolated over a 24 hour period.

The LYSSY method provides a higher MVTR value than ASTM E96, Method B for a permeable fabric like the flash-spun sheet material of the invention. Use of the two methods highlights the differences in MVTR measurements that can result from using different test methods.

Gurley Hill Porosity is a measure of the air permeability of the sheet material for gaseous materials. In particular, it is a measure of how long it takes for a volume of gas to pass through an area of material wherein a certain pressure gradient exists. Gurley-Hill porosity is measured in accordance with TAPPI T-460 om-88 using a Lorentzen & Wettre Model 121D Densometer. This test measures the time of which 100 cubic centimeters of air is pushed through a one inch diameter sample under a pressure of approximately 4.9 inches of water. The result is expressed in seconds and is usually referred to as Gurley Seconds.

Frazier Porosity is a measure of air permeability of porous materials and is reported in units of $\text{ft}^3/\text{ft}^2/\text{min}$. It measures the volume of air flow through a material at a differential pressure of 0.5 inches water. An orifice is mounted in a vacuum system to restrict flow of air through sample to a measurable amount. The size of the orifice depends on the porosity of the material. Frazier porosity is measured using a Sherman W. Frazier Co. dual manometer with calibrated orifice units $\text{ft}^3/\text{ft}^2/\text{min}$.

Elongation to Break of a sheet is a measure of the amount a sheet stretches prior to failure (breaking) in a strip tensile test. A 1.0 inch (2.54

cm) wide sample is mounted in the clamps - set 5.0 inches (12.7 cm) apart - of a constant rate of extension tensile testing machine such as an Instron table model tester. A continuously increasing load is applied to the sample at a crosshead speed of 2.0 in/min (5.08 cm/min) until failure. The measurement is given in percentage of stretch prior to failure. The test generally follows ASTM D1682-64.

Opacity relates to how much light is permitted to pass through a sheet. One of the qualities of Tyvek® sheet is that it is opaque and one cannot see through it. Opacity is the measure of how much light is reflected or the inverse of how much light is permitted to pass through a material. It is measured as a percentage of light reflected. Although opacity measurements are not given in the following data tables, all of the examples have opacity measurements above 90 percent and it is believed that an opacity of at least about 85 is minimally acceptable for almost all end uses.

Handle-o-meter Stiffness is a measure of the resistance of a specimen from being pressed into a 10 mm slot using a 40 gm pendulum. It is measured by INDA IST 90.3-92. As one would expect, the stiffness tends to increase with basis weight. Thus, the stiffness has been normalized by the basis weight.

Moisture permeability index ("im") is defined as the ratio of the thermal and evaporative resistance of a fabric to the ratio of thermal and evaporative resistance of air (theoretical limit). It is calculated from the wet and dry heat transfer properties measured using the Thermolabo II "Sweating Hot Plate" Method developed by Kawabata et.al., which is described in: Kawabata et al., "Application of the New Thermal Tester THERMOLABO to the Evaluation of Clothing Comfort," Objective Measurement: Application to Product Design and Process Control, The Machinery Society of Japan, 1985, which is hereby incorporated by reference.

Thermal resistance ("clo" units) is a measure of clothing insulation. The dry heat transfer property of a fabric is measured using the Thermolabo II "Sweating Hot Plate" method per Kawabata et.al. This value

can be converted into the more conventional "clo" units of clothing insulation.

Theoretical Activity Limits ("comfort" and "maximum") are calculated by inputting the "clo" and "im" values along with specified environmental conditions (T_{amb} , P_{amb}) into a heat balance equation as developed by A.H. Woodcock. Woodcock's equation is more fully described in: Woodcock, A.H., "Moisture in Textile Systems, Parts I and II," Textile Research Journal, 32, 1962, pp. 626, 719, which is hereby incorporated by reference. The comfort limit uses a factor to allow for a sweat wetted area of 20% (determined to be the comfort limit for the human wearer) and a sweat wetted area of 100%, fully wetted condition to be the maximum limit (beyond which there is no longer thermoregulation).

Bacteria Spore Penetration is measured according to ASTM F1608-95, which is hereby incorporated by reference. According to this method, a sheet sample is exposed to an aerosol of bacillus subtilis var. niger spores for 15 minutes at a flow rate through the sample of 2.8 liters/min. Spores passing through the sample are collected on a media and are cultured and the number of cluster forming units are measured. The % penetration is the ratio of the cluster forming units measured on the media downstream of the sample versus the number of cluster forming units obtained on a media where no sheet sample was present.

Filtration Efficiency is measured according to IES standard IES-RP-CC003.2, Section 7.3.1 (Garment System Considerations for Cleanroom and Other Controlled Environments; Particle Penetration), which is hereby incorporated by reference. According to this method, five samples are mounted in a 25 cm diameter filter holder. A vacuum pump is used to establish a flow of ambient room air through the fabric at a rate that yields a pressure drop of 1 cm of water. A Climet Instruments Model 226/8040 aerosol analyzer is used to obtain 10 1-minute upstream and 10 1-minute downstream counts of particles with diameters greater than 0.5 microns. This data is used to calculate the filtration efficiency according to the following formula:

$$\text{Efficiency} = 1 - \frac{\text{avg. downstream particle count}}{\text{avg. upstream particle count}}$$

- 5 Penetration Velocity is a product of the penetration and the face velocity and is calculated in units of cm/min from the Filtration Efficiency data as follows:

$$\text{Penetration Velocity} = \frac{(\text{avg. downstream particle count}) (\text{volumetric flow rate})}{(\text{avg. upstream particle count}) (\text{filtration area})}$$

10

EXAMPLES 1-8

- In the Examples 1-8, nonwoven sheets were flash-spun from high density polyethylene with a melt index of 0.70 g/10 minutes (@ 190° C with a 2.16 kg weight), a melt flow ratio {MI (@ 190° C with a 2.16 kg weight)/MI (@ 190° C with a 21.6 kg weight)} of 34, and a density of 0.96 g/cc. The sheets were flash-spun according to the process described above under one of two spin conditions. Under Condition A, the spin solution comprised of 88% FREON®-11 and 12% high density polyethylene, and the spinning temperature was 180°C. Under Condition B, the spin solution comprised 84% n-pentane and 16% high density polyethylene, and the spinning temperature was 175°C. The sheets of Examples 2, 4, 6 and 8 were produced under condition A, and the sheets of Examples 1, 3, 5, and 7 were produced under Condition B. Sheet samples produced under Condition A were paired with samples produced under Condition B, and four such sample pairs were bonded on the same 34" thermal bonder using a linen and "P" point pattern without mechanical softening. The samples of each sample pair were subjected to the same bonding conditions. The bonding conditions and sheet properties are reported in Table 1, below.
- 15
- 20
- 25

TABLE 1

<u>Spinning Condition</u>	<u>Ex. 1</u> B	<u>Ex. 2</u> A	<u>Ex. 3</u> B	<u>Ex. 4</u> A
<u>Bonding Conditions</u>				
Steam Pressure (kPascal-gauge)	385	385	440	440
Bonding Temp. (°C)	131	133	~136	136
Nip Pressure (kPascal)	3450	3450	3450	3450
<u>Comfort/Barrier Properties</u>				
MVTR (g/m ² /day)	1079	710	1119	745
MVTR-LYSSY (g/m ² /day)	-	-	-	-
Hydrostatic Head (cm)	185	163	203	142
<u>Other Physical Properties</u>				
Basis Weight (g/m ²)	42.0	42.4	41.7	42.4
Delamination (N/m)	12.5	10.5	14	12.5
Crock Meter - Linen Side (# of Strokes)	2	7	3	3
Crock Meter - "P" Side (# of Strokes)	11	4	17	6
Tensile Strength MD (N/m)	1600	1250	1600	1250
Tensile Strength XD (N/m)	1750	1750	2100	1600
Elongation MD (%)	13	8	14	8
Elongation XD (%)	18	13	19	14
Tongue Tear MD (N/m)	550	550	550	550
Tongue Tear XD (N/m)	550	550	550	550
Thickness (μm)	130	137	122	142
Density (g/cm)	0.323	0.309	0.342	0.299

TABLE 1 (continued)

<u>Spinning Condition</u>	<u>Ex. 5</u> B	<u>Ex. 6</u> A	<u>Ex. 7</u> B	<u>Ex. 8</u> A
<u>Bonding Conditions</u>				
Steam Pressure (kPascal)	470	470	485	485
Bonding Temp. (°C)	136	137	139	137
Nip Pressure (kPascal)	3450	3450	5515	5515
<u>Comfort/Barrier Properties</u>				
MVTR (g/m ² /day)	1174	802	910	541
MVTR-LYSSY (g/m ² /day)	1139	926	1035	-
Hydrostatic Head (cm)	198	160	238	172
<u>Other Physical Properties</u>				
Basis Weight (g/m ²)	41.4	43.1	41.0	42.7
Delamination (N/m)	14	12.5	19.5	14
Crock Meter - Linen Side (# of Strokes)	3	11	19	19
Crock Meter - "P" Side (# of Strokes)	18	2	21	14
Tensile Strength MD (N/m)	1600	1400	2300	2100
Tensile Strength XD (N/m)	2100	1750	2650	2450
Elongation MD (%)	13	10	16	14
Elongation XD (%)	22	14	19	16
Tongue Tear MD (N/m)	550	350	350	350
Tongue Tear XD (N/m)	550	550	550	350
Thickness (μm)	130	155	107	130
Density (g/cm)	0.318	0.278	0.383	0.328

Under each of the four bonding conditions in Examples 1-8, a
 5 dramatic improvement in MVTR can be seen when the sheet produced under

the the new hydrocarbon based spinning conditions (Condition B) is compared against sheet produced under conventional FREON®-11 manufacturing conditions (Condition A). Importantly, these MVTR improvements are in each side by side comparison accompanied by a modest increase in liquid barrier. The MVTR of the Condition B samples were on average 54.2% better than that of the samples spun under Condition A. This is especially significant because the liquid barrier (Hydrohead) offered by the new more air permeable material produced according to Condition B is on average about 30% greater than the liquid barrier provided by the conventional samples spun under Condition A. When one compares samples of the old product (Condition A) and the new product (Condition B) having the same delamination strength (meaning that the sheets are bonded to the same degree but not necessarily under the same bonding conditions) such as Examples 5 and 8 above, the MVTR improvements become more pronounced while the Hydrostatic Head maintains a substantial improvement.

Examples 9-15

In the Examples 9-15, nonwoven sheets were flash-spun from the high density polyethylene of Examples 1-8. The sheets were spun as described above from a spin solution comprised n-pentane and high density polyethylene. The flash-spinning conditions were varied by changing the concentration of the polymer in the spin solution and by altering the spinning temperature. The sheets were all thermal bonded using a linen and "P" point pattern under the same conditions (bonding pressure of 5515 kPa (800 psi) on a 34" bonding calendar with steam pressure at 483 kPa-gauge (70 psig), and without mechanical softening). The polymer concentration and spin solution temperature used in making each sample and the properties of the samples are reported in Table 2, below.

TABLE 2

	<u>Ex. 9</u>	<u>Ex. 10</u>	<u>Ex. 11</u>	<u>Ex. 12</u>
<u>Spinning Conditions</u>				
Concentration (%)	22	18	16	16
Solution Temp. (°C)	175	189	175	185
<u>Comfort/Barrier Properties</u>				
MVTR (g/m ² /day)	1201	1306	1038	1330
MVTR-LYSSY (g/m ² /day)	1204	1470	1235	1554
Hydrostatic Head (cm)	79	163	203	201
Gurley Hill Porosity (seconds)	52	89	339	77
<u>Other Physical Properties</u>				
Basis Weight (g/m ²)	40.5	40.5	40.5	40.5
Delamination (N/m)	24.5	10.5	24.5	26.5
Crock Meter - Linen Side (# of Strokes)	25	15	22	20
Crock Meter - "P" Side (# of Strokes)	20	10	25	16
Tensile Strength MD (N/m)	1600	1950	2300	1750
Tensile Strength XD (N/m)	1950	2100	2650	1600
Elongation MD (%)	14	16	15	17
Elongation XD (%)	23	22	20	25
Work to Break MD (N-m)	0.6	0.7	0.8	0.7
Work to Break XD (N-m)	0.9	0.9	1.0	0.8
Tongue Tear MD (N/m)	350	350	350	350
Tongue Tear XD (N/m)	550	350	550	350

TABLE 2 (continued)

	<u>Ex. 13</u>	<u>Ex. 14</u>	<u>Ex. 15</u>
<u>Spinning Conditions</u>			
Concentration (%)	14	14	12
Solution Temp. (°C)	175	184	175
<u>Comfort/Barrier Properties</u>			
MVTR (g/m ² /day)	1175	1333	1245
MVTR-LYSSY (g/m ² /day)	1243	1368	1389
Hydrostatic Head (cm)	175	232	196
Gurley Hill Porosity (seconds)	200	84	161
<u>Other Physical Properties</u>			
Basis Weight (g/m ²)	44	40.5	40.5
Delamination (N/m)	23	24.5	61.5
Crock Meter - Linen Side (# of Strokes)	25	25	25
Crock Meter - "P" Side (# of Strokes)	24	24	25
Tensile Strength MD (N/m)	1750	1950	1950
Tensile Strength XD (N/m)	1950	2300	2300
Elongation MD (%)	27	23	29
Elongation XD (%)	39	37	49
Work to Break MD (N-m)	1.0	1.0	1.2
Work to Break XD (N-m)	1.5	1.2	1.5
Tongue Tear MD (N/m)	350	350	175
Tongue Tear XD (N/m)	350	350	175

Examples 9-15 demonstrate that excellent MVTR can be achieved at a variety of polymer concentrations when plexifilamentary sheet material is flash spun from a hydrocarbon-based spin agent, even in the absence of mechanical softening. The Gurley Hill Porosity values for Examples 9-15 would be expected to be substantially lower if mechanical softening were

present. In addition, Example pairs 11-12 and 13-14 show that increasing the solution spin temperature while keeping the polymer concentration constant also results in a dramatic improvement in both MVTR and Gurley Hill porosity, without any significant loss in liquid barrier properties.

5

Examples 16-21

In the Examples 16-21, nonwoven sheets were flash-spun from the high density polyethylene of Examples 1-8. The sheets were spun as described above from a spin solution comprised FREON®-11 and high
10 density polyethylene. The flash-spinning conditions were varied by changing the concentration of the polymer in the spin solution and by altering the spinning temperature. The sheets were all thermally bonded (rib and linen pattern) and softened at commercial conditions similar to those used for conventional 1.2 oz/yd² TYVEK® used in the protective apparel
15 market. The oil temperature range for the rib and linen embossers was 160°-190° C and the pin roll penetration for softening was 0.045 inch (1.14 cm). The polymer concentration and spin solution temperature used in making each sample and the properties of the samples are reported in Table 3, below.

TABLE 3 (continued)

	<u>Ex. 19</u>	<u>Ex. 20</u>	<u>Ex. 21</u>
<u>Spinning Conditions</u>			
Concentration (%)	10	10	9
Spin Temp. (°C)	189	195	189
<u>Comfort/Barrier Properties</u>			
MVTR-LYSSY (g/m ² /day)	1546	1575	1463
MVTR (g/m ² /day)	-	-	1438
Hydrostatic Head (cm)	131	124	188
Gurley Hill Porosity (seconds)	13	9	11
<u>Other Physical Properties</u>			
Basis Weight (g/m ²)	40.7	40.7	41.0
Delamination (N/m)	11	12	14
Tensile Strength MD (N/m)	1408	1658	1450
Tensile Strength XD (N/m)	1564	1487	1750
Elongation MD (%)	10.54	9.43	10.6
Elongation XD (%)	16.93	15.61	17.5
Work to Break MD (N-m)	0.305	0.325	0.33
Work to Break XD (N-m)	0.487	0.400	0.60
Tongue Tear MD (N/m)	-	352	260
Tongue Tear XD (N/m)	349	401	330

Examples 16-21 demonstrate that when flash-spinning sheet material from a FREON®-based spin solution, MVTR can be improved, without any significant loss in liquid barrier (hydrohead), by increasing the spin solution temperature while the polymer concentration is held constant. Importantly, the results in Examples 16-21 also demonstrate that fabrics with improved MVTR and Gurley Hill porosity properties can be obtained using a FREON®-based spin solution, as compared to the MVTR and Gurley Hill

porosity properties of sheets made using the conventional 12% polymer concentration and 180° C spin temperature (*see* Examples 22 and 39).

Examples 22-27

5 In Examples 22-27, samples of flash-spun polyethylene sheet material made according to a variety of process conditions were tested according to a number of comfort indicators. In Examples 22-27, a nonwoven sheet was flash-spun from the high density polyethylene of Examples 1-8. The sheet was spun as described above from a spin solution
10 of high density polyethylene in a solvent that was either FREON®-11 ("F") or n-pentane hydrocarbon ("H"). The sheets were bonded as described below. The polymer concentration (weight % of solution) and spin solution temperature used in making each sample and certain comfort properties of the samples are reported in Table 4, below.

15 The samples in Examples 22 and 23 were produced under the same conditions except that an inline static mixer (*see* Figure 2, #36) was inserted in the letdown chamber during spinning in Example 23, but not in Example 22. In each sample, the sheet was thermally bonded (rib and linen pattern) and softened at commercial conditions similar to those used for
20 conventional 1.2 oz/yd² TYVEK® used in the protective apparel market. The oil temperature range for the rib and linen embossers was 160°-190° C and the pin roll penetration for softening was 0.045 inch (1.14 cm).

 The samples in Examples 24, 26 and 27 were point bonded on a 34" laboratory thermal bonder under duplicate conditions using a linen and
25 "P" point pattern and they were not mechanically softened. Example 25 was point bonded under the bonding conditions described in the paragraph above with respect to Examples 22 and 23.

 Example 26 corresponds to Example 11 above. Example 27 corresponds to Example 12 described above.

30

Table 4

	<u>Ex. 22</u>	<u>Ex. 23</u>
<u>Spinning/Bonding Conditions</u>		
Solvent	F	F
Polymer Concentration (%)	12	12
Solution Temperature (°C)	180	180
Thermal Point Bonding?	Yes	Yes
Mechanical Softening?	Yes	Yes
Static Mixer?	No	Yes
<u>Comfort/Barrier Properties</u>		
IM	0.16	0.235
CLO	0.425	0.417
Comfort Limit	245	265
Max Limit	373	457
Hydrostatic Head (cm)	-	127
MVTR (g/m ² /day)	1070	1327
Gurley Hill Porosity (sec)	25.2	9.15
<u>Other Properties</u>		
Thickness (mm)	0.23	0.27
Basis Weight (g/m ²)	42.7	41.4

- 5 Examples 22 and 23 demonstrated that the addition of a static mixer in the letdown chamber improves both MVTR and Gurley Hill porosity, as well as relative comfort as indicated by the Comfort and Max Limit values.

Table 4 (Continued)

	<u>Ex. 24</u>	<u>Ex. 25</u>	<u>Ex. 26</u>	<u>Ex. 27</u>
<u>Spinning/Bonding Conditions</u>				
Solvent	F	F	H	H
Polymer Concentration (%)	12	11	16	16
Solution Temperature (°C)	180	186	175	185
Thermal Point Bonding?	Yes	Yes	Yes	Yes
Mechanical Softening?	No	Yes	No	No
<u>Fiber Size Distribution</u>				
Mean	18.2	11.0	12.6	13.3
Standard Deviation	19.6	10.9	9.0	12.0
<u>Comfort/Barrier Properties</u>				
IM	0.102	0.299	0.125	0.258
CLO	0.408	0.421	0.381	0.401
Comfort Limit	243	276	265	280
Max Limit	328	518	378	500
Hydrostatic Head (cm)	172	152	203	201
MVTR (g/m ² /day)	541	1419	1038	1330
Gurley Hill Porosity (sec)	>180	11.1	339	77
<u>Other Properties</u>				
Thickness (mm)	0.13	0.37	0.17	0.21
Basis Weight (g/m ²)	42.7	43.1	40.7	41.7

- In the foregoing examples it should be noted that the lower
- 5 concentration higher temperature samples had smaller fiber sizes which has apparently translated to dramatically increased MVTR and substantially improved permeability (fewer Gurley seconds). The CLO, IM, Comfort Limit and Max Limit values also predict that the finer fiber sheet material of Examples 25 and 27 will offer workers wearing protective apparel made

from the new finer fiber fabric more comfort without a significant reduction in liquid barrier properties.

Examples 28-30

- 5 In the following Examples 28-30, competitive materials used in protective apparel have been compared using the same testing facilities and procedures as used in Examples 22-27 above. In particular, Example 28 is a microporous film spunbonded laminate. Example 29 is another microporous film spunbonded laminate. Example 30 is a
- 10 spunbonded/meltblown/spunbonded ("SMS") composite.

TABLE 5

	<u>Ex. 28</u>	<u>Ex. 29</u>	<u>Ex. 30</u>
<u>Comfort/Barrier Properties</u>			
IM	0.238	0.418	0.402
CLO	0.45	0.478	0.392
Comfort Limit	246	264	318
Max Limit	427	563	669
Hydrostatic Head (cm)	>399*	>399*	53
MVTR (g/m ² /day)	1191	1506	1766
Gurley Hill Porosity (sec)	>100	>100	90 ⁺
<u>Other Properties</u>			
Thickness (mm)	0.57	0.41	0.44
Basis Weight (g/m ²)	64.4	57.0	50.9

- * - these products had to be provided with a supporting scrim to
- 15 prevent delamination of the film. The maximum measurement capability of the equipment is 399 cm (157 inches) and, once supported with the scrim, these materials surpassed the equipment limits.

⁺SMS air permeability measured in Frazier Porosity cfm/ft²

From Examples 28 and 29, it can be seen that the flash-spun sheet material of the invention has achieved MVTR, IM, Clo, and predicted Comfort Limit values comparable to microporous films, and Gurley Hill porosity values far superior to that of microporous films. Example 30 demonstrates that SMS materials have excellent comfort properties. However, as will be evident from Examples 37 and 40 below, SMS offers a wearer relatively little barrier protection.

Examples 31-35

10 In the Examples 31-35, nonwoven sheets were flash-spun from the high density polyethylene of Examples 1-8. The sheets were spun as described above from a spin solution comprised of FREON®-11 and high density polyethylene. The flash-spinning conditions were varied by changing the concentration of the polymer in the spin solution and by
15 altering the spinning temperature. The sheets were thermally bonded (rib and linen pattern) and softened at commercial conditions similar to those used for conventional 1.2 oz/yd² TYVEK® used in the protective apparel market. The oil temperature range for the rib and linen embossers was 160°-190° C and the pin roll penetration for softening was 0.045 inch
20 (1.14 cm). The sheets were tested for bacterial spore penetration. The polymer concentration and spin solution temperature used in making each sample and the properties of the samples are reported in Table 6, below. The sample in Example 37 is a competitive spunbonded/meltblown/spunbonded ("SMS") material for use in protective garments.

TABLE 6

	<u>Ex. 31</u>	<u>Ex. 32</u>	<u>Ex. 33</u>	<u>Ex. 34</u>
<u>Spinning Conditions</u>	(control)			
Concentration (%)	12	11	11	10
Solution Temp. (°C)	180	180	190	180
<u>Properties</u>				
Spore Penetration (%)	0.10	0.04	0.05	0.06
Penetration Std Deviation (n)	0.14 (10)	0.06 (9)	0.05 (10)	0.08 (9)
MVTR LYSSY (g/m ² /day)	1017	1153	1516	1488
Gurley Hill Porosity (seconds)	31	5.7	~8.2	Not avail

* n is the number of specimens per material sample.

5

TABLE 6 (continued)

	<u>Ex. 35</u>	<u>Ex. 36</u>	<u>Ex. 37</u>
<u>Spinning Conditions</u>			SMS
Concentration (%)	10	10	-
Solution Temp. (°C)	185	195	-
<u>Properties</u>			
Bacteria Spore Penetration (%)	0.02	0.07	54.5
Penetration Std Deviation (n)	0.03 (10)	0.13 (10)	14.5(10)
MVTR LYSSY (g/m ² /day)	1453	1329	61*
Gurley Hill Porosity (seconds)	~11.0	~7.6	NA

* SMS air permeability measured in Frazier Porosity cfm/ft²

10 Examples 31-36 demonstrate that the composite sheet material of the invention (Examples 32-36), as compared to conventional Tyvek® sheet material used in protective apparel (Ex. 31), offers at least equivalent barrier to bacteria penetration while offering substantially improved MVTR and air

permeability (lower Gurley seconds). Example 37 shows that the competitive SMS product offers far less barrier to bacteria penetration than is afforded by the sheet material of Examples 31-36.

5

Examples 38-40

In the Examples 38 and 39, nonwoven sheets were flash-spun from the high density polyethylene of Examples 1-8. The sheets were spun as described above from a spin solution comprised of FREON®-11 and high density polyethylene. The flash-spinning conditions were varied by
10 changing the concentration of the polymer in the spin solution and by altering the spinning temperature. The sheets were thermally bonded (rib and linen pattern) and softened at commercial conditions similar to those used for conventional 1.2 oz/yd² TYVEK® used in the protective apparel market. The oil temperature range for the rib and linen embossers was
15 160°-190° C and the pin roll penetration for softening was 0.045 inch (1.14 cm). The polymer concentration and spin solution temperature used in making each sample and the properties of the samples are reported in Table 7 below.

The sample in Example 38 is the fine fiber material of the present
20 invention. The sample in Example 39 is a piece of conventional Tyvek® Type 1422A sheet material used in protective garments. The sample in Example 40 is a competitive spunbonded/meltblown/spunbonded ("SMS") material for use in protective garments.

TABLE 7

	<u>Ex. 38</u>	<u>Ex. 39</u>	<u>Ex. 40</u>
<u>Spinning Conditions</u>			
Concentration (%)	11	12	NA
Solution Temp. (°C)	190	180	NA
<u>Properties</u>			
Filtration Efficiency (%)	98.0	94.2	50.8
Efficiency Std Deviation	0.4	1.9	2.2
Avg. Penetration Velocity (cm/min)	0.40	0.91	1,034
MVTR LYSSY (g/m ² /day)	1509	1334	1766
Gurley Hill Porosity (seconds)	4.5	18.7	74.2*

* SMS air permeability measured in Frazier Porosity cfm/ft²

5

Examples 38 and 39 demonstrate that the sheet material of the invention (Ex. 38), as compared to conventional Tyvek® sheet material used in protective apparel (Ex. 39), offers at least equivalent barrier to dry particulates while offering substantially improved MVTR and air permeability (lower Gurley seconds). Example 40 shows that the competitive SMS product offers far less barrier to dry particulate penetration than is afforded by the sheet material of Examples 38 and 39.

10

The foregoing description and drawings were intended to explain and describe the invention so as to contribute to the public base of knowledge. In exchange for this contribution of knowledge and understanding, exclusive rights are sought and should be respected. The scope of such exclusive rights should not be limited or narrowed in any way by the particular details and preferred arrangements that may have been shown. Clearly, the scope of any patent rights granted on this application should be measured and determined by the claims that follow.

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WE CLAIM:

1. A synthetic sheet material useful in protective apparel, said sheet material having a hydrostatic head pressure of at least about 75 cm of water, a Gurley Hill Porosity of less than about 15 seconds, and a
5 Handle-o-meter stiffness of less than 28 mN/g/m².
2. The sheet material of claim 1 wherein the sheet material has a hydrostatic head pressure of at least about 90 cm of water and a Gurley Hill Porosity of less than about 12 seconds.
3. The sheet material of claim 2 wherein the sheet material
10 has a Gurley Hill Porosity of less than about 10 seconds.
4. The sheet material of claim 1 wherein the sheet material has a bacteria spore penetration, measured according to ASTM F1608-95, of less than 5%.
5. The sheet material of claim 4 wherein the sheet material
15 has a bacteria spore penetration, measured according to ASTM F1608-95, of less than 1%.
6. The sheet material of claim 1 wherein the sheet material has a filtration efficiency, measured according to IES standard IES-RP-CC003.2, Section 7.3.1, of at least 95%.
- 20 7. The sheet material of claim 1 wherein the sheet material has an average penetration velocity, measured according to IES standard IES-RP-CC003.2, Section 7.3.1, of less than 1.0 cm/min.
8. The sheet material of claim 2 wherein the sheet material has an MVTR-LYSSY, measured according to ASTM E398-83, of at least
25 1300 g/m²/day.
9. The sheet material of claim 2 wherein the sheet material has a basis weight of at least 30 g/m².

10. The sheet material of claim 1 wherein the sheet material has a tensile strength in both the machine and cross directions of at least 1250 N/m.
- 5 11. The sheet material of claim 1 wherein the sheet material has a tongue tear in both the machine and cross directions of 250 N/m.
12. A synthetic sheet material comprised primarily of nonwoven fibers, said sheet material having a hydrostatic head pressure of at least about 75 cm of water, a Gurley Hill Porosity of less than about 15 seconds, and a Handle-o-meter stiffness of less than 28 mN/g/m².
- 10 13. The sheet material of Claim 12 wherein the sheet material is substantially exclusively nonwoven fibers.
14. The sheet material of Claim 13 wherein the sheet material is comprised of a unitary sheet of nonwoven fibers.
- 15 15. The sheet material of Claim 12 wherein said nonwoven fibers are flash-spun plexifilamentary fibrils comprised of polyolefin polymer.
16. The sheet material of claim 15 wherein said polyolefin is high density polyethylene.
- 20 17. The sheet material of Claim 12 wherein the basis weight of the sheet material is above about 35 g/m².
18. The sheet material of Claim 17 wherein the sheet material has an MVTR-LYSSY, measured according to ASTM E398-83, of at least about 1350 g/m²/day.
- 25 19. A protective garment comprising a plurality of interconnected sheet material pieces, each of said sheet material pieces comprising a synthetic sheet material having a hydrostatic head pressure of at least about 75 cm of water, a Gurley Hill Porosity of less than about 15 seconds, and a Handle-o-meter stiffness of less than 28 mN/g/m².